Materials Science

15A/2007G508

Synthesis of Silver Particles in Tween20/Water/Ionic Liquid Microemulsions

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Introduction

Room-temperature ionic liquids (RTILs) are attracting much interest in many fields of chemistry and industry. Because of their unique physical and chemical properties, the latest developments of RTILs as a reaction medium for inorganic nanomaterials have received much attention and could provide many opportunities and challenges especially for the synthesis of nanoparticles with the unique shape and structures [1, 2]. In this study we have synthesized Ag particles by the photoreduction of silver perchlorate ($AgClO_4$) in the microemulsions constituted of Tween20/water/1-octyl-3-methylimidazolium tetrafluoroborate ([omim][BF_4])/CO_2 system. We have also investigated the size of Ag particles as well as the water droplets containing Ag particles in the emulsions by *in-situ* SAXS measurements. Here we have applied the high pressure CO_2 to the ternary system in order to improve the mobility of ions and particles.

Experimental

Ag particles were synthesized in a high-pressure SUS 316 cell (inner volume of 13.5 mL) equipped with four optical windows: two of them were diamond windows for *in-situ* SAXS measurements and the other two were quartz windows for the UV-irradiation of mercury lamp. Tween20/water/[omim][BF_4] microemulsions containing $AgClO_4$ were prepared by adding CO_2 into the cell which contained a mixture of $AgClO_4$ aqueous solution, Tween20, [omim][BF_4], and benzoin for photoreduction. The weight fraction of Tween20 was 0.33, and the [omim][BF_4]-to-Tween20 molar ratio (R) and the water-to-Tween20 molar ratio (w) was fixed to 8.7 and 0.62, respectively. Both concentrations of [Ag^+] and benzoin in the ternary system were 4.4 mM. The cell was kept at 35°C and 25MPa for 1 h with continuous stirring to form microemulsions. After stirring, the microemulsion was photo-irradiated for the designated time to investigate the reduction of Ag^+ and the formation of Ag particles. The *in-situ* SAXS measurements were performed at BL-15A. The scattering data was collected by a position sensitive proportional counter (PSPC).

Results and Discussion

Figure 1(a) shows the SAXS profiles of Tween20/water/[omim][BF_4]/CO_2 microemulsions containing Ag^+ ions before and after the photoreduction for the designated time. The intensity at a small q range ($q < 1.5$ nm^{-1}) increases with an increase of reduction time.

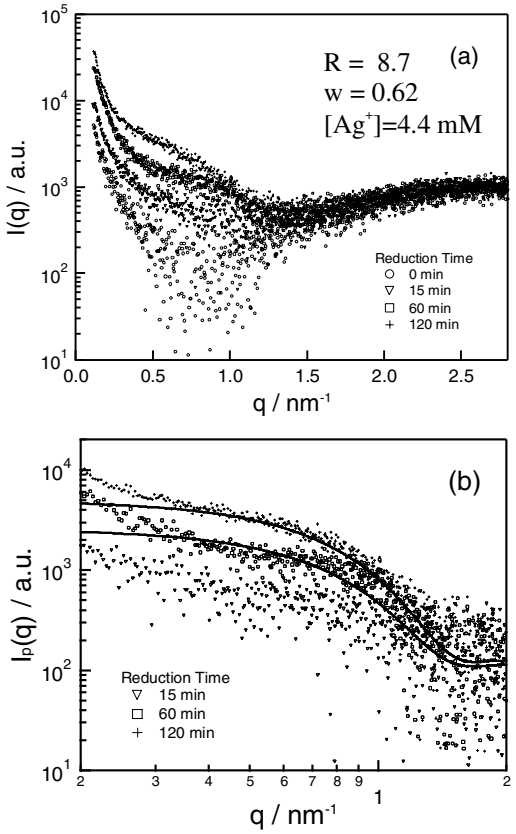

Fig. 1. (a) SAXS profiles of the Ag solutions before and after the photoreduction and (b) time evolution of intensity from Ag particles. Solid lines show the best-fitted curves theoretically obtained with the Schultz-Zimm distribution.

The increase of the scattering intensity indicates the formation of Ag particles during the UV-irradiation. To analyze the size distribution of Ag particles, the scattering intensity from the Ag particles ($I_p(q)$) is separated from the observed scattering intensity ($I(q)$) by subtracting the intensity of the microemulsions containing Ag^+ ions before UV-irradiation, as shown in Fig. 1(b). By using the Schultz-Zimm distribution, the number-averaged particle radius of the Ag particles is determined as 2.8 nm (reduction time of 120 min), which is consistent with that obtained from TEM observation.

References

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